

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 11-084638

(43)Date of publication of application : 26.03.1999

(51)Int.Cl.

G03F 7/004
C09D 4/00
C09D 5/00
C09D201/00
H01J 9/02
// C08F299/00

(21)Application number : 09-246972

(71)Applicant : TORAY IND INC

(22)Date of filing : 11.09.1997

(72)Inventor : YAMASHITA SHINICHI
IGUCHI YUICHIRO
MASAKI YOSHIKI

(54) PHOTSENSITIVE GLASS PASTE AND PRODUCTION OF PLASMA DISPLAY

(57)Abstract:

PROBLEM TO BE SOLVED: To form a paste coating layer having a uniform thickness by specifying the b.p. difference between at least two or more kinds of org. solvents contained in a photosensitive glass paste.

SOLUTION: The photosensitive glass paste consists of glass powder, a photosensitive org. component and two or more org. solvents and the b.p. difference between at least two of the org. solvents is $\geq 20^{\circ}$ C. The amts. of the low and high b.p. org. solvents are 10-90 wt.% each of the total amt. of the org. solvents. The total amt. of the org. solvents is 1-50 wt.% of the amt. of the entire photosensitive glass paste. An oligomer or polymer having carboxyl groups and/or unsatd. double bonds in one molecule and a wt. average mol.wt. of 500-100,000 is essentially contained in the photosensitive org. component by 10-90 wt.%.

LEGAL STATUS

[Date of request for examination] 19.02.2002

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] 3520733

[Date of registration] 13.02.2004

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

* NOTICES *

Japan Patent Office is not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.*** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] The photosensitive glass paste which contains these two or more kinds of organic solvents, and is characterized by the boiling point difference of at least two kinds of organic solvents being 20 degrees C or more among these organic solvents in the photosensitive glass paste which consists of glass powder, a photosensitive organic component, and an organic solvent.

[Claim 2] The photosensitive glass paste according to claim 1 characterized by the organic solvent by the side of a low-boiling point and a high-boiling point containing ten to 90% of the weight to the organic solvent whole quantity among said organic solvents, respectively.

[Claim 3] Claim 1 characterized by the total content of said organic solvent being in 1 - 50% of the weight of the range to the whole photosensitive glass paste, or a photosensitive glass paste according to claim 2.

[Claim 4] The ~~*****~~-strike according to claim 1 to 3 characterized by containing in intramolecular the oligomer or the polymer of the weight molecular weight 500-100,000 which has a partial saturation double bond ten to 90% of the weight to the amount of photosensitive organic substance as an indispensable component of said photosensitive organic component at a carboxyl group or/and intramolecular.

[Claim 5] The manufacture approach of the plasma display which is the manufacture approach of a plasma display including the process which applies on a substrate, is pattern-exposed, develops a photosensitive glass paste according to claim 1 to 4 after desiccation, calcinates further, and forms a septum, and is characterized by setting 1 time of spreading thickness of this photosensitive glass paste to 50 micrometers or more.

[Claim 6] The manufacture approach of the plasma display according to claim 5 characterized by applying this photosensitive glass paste that forms said septum by the doctor blade method or the slit-die coat method.

[Claim 7] The manufacture approach of the plasma display according to claim 5 or 6 characterized by applying this photosensitive glass paste that forms said septum by 1 time or 2 times.

[Translation done.]

* NOTICES *

Japan Patent Office is not responsible for any damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.

2.**** shows the word which can not be translated.

3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the manufacture approach of the plasma display using the photosensitive glass paste and it which are used for a plasma display, a plasma-address-liquid-crystal display, etc.

[0002]

[Description of the Prior Art] Since a high-speed display is possible for a plasma display panel (PDP) compared with a liquid crystal panel and it is easy to enlarge, it has permeated the field of OA equipment, a public-relations display, etc. Furthermore, progress in the field of a high definition television etc. is expected dramatically.

[0003] With such application amplification, it is delicate and the color PDP which has many display cells attracts attention. This color PDP makes the anode and the cathode inter-electrode which oppose in the discharge space which it had between the glass front substrate and the tooth-back glass substrate produce plasma discharge, and displays by hitting against the fluorescent substance which established the ultraviolet rays generated from the gas enclosed in the above-mentioned discharge space in discharge space. In this case, in order to secure uniform discharge space, the septum (it is also called an obstruction or a rib) is formed at the same time it makes a presser foot and a display perform within the cell of a convention of the breadth of discharge in a fixed field. The configuration of this septum is 60-200 micrometers in width of face of 20-80 micrometers, and height about with a high definition PDP, and this septum applies the *****-strike which becomes a glass front substrate and a tooth-back glass substrate from glass, and is formed through desiccation, exposure, development, and baking.

[0004] Screen printing is known as an approach of forming a septum layer. However, by this approach, since 1 time of spreading thickness is dozens of micrometers, in order to form a septum layer with a height of 60-200 micrometers, generally it is necessary to repeat printing/desiccation 10 times or more many times, and there is a trouble that productivity is very bad. Then, in order to solve this problem, there is the approach of applying by the doctor blade method or the slit-die coat method as an approach of applying a paste at once. However, after especially applying the paste by doctor blade spreading in this 1-time spreading and making it dry, about 30 micrometers of edge sections of a paste coating layer (septum formative layer) rose, and there was a trouble that the spreading layer of uniform thickness could not be formed.

[0005]

[Problem(s) to be Solved by the Invention] There is little climax of the edge section of a paste coating layer, and this invention aims at offering the photosensitive glass paste which can form the paste coating layer of uniform thickness, when a photosensitive glass paste is applied and this is dried.

[0006] Other objects of this invention are to offer the manufacture approach of a quality plasma display of having used this photosensitive glass paste.

[0007]

[Means for Solving the Problem] In order to solve the above-mentioned technical problem, in the photosensitive glass paste which consists of glass powder, a photosensitive organic component, and an organic solvent, the photosensitive glass paste of this invention is characterized by containing these two or more kinds of organic solvents, and the boiling point difference of at least two kinds of organic solvents considering as 20 degrees C or more among these organic solvents, and includes the following desirable embodiment further by this invention.

[0008] (a) The content of the organic solvent by the side of a low-boiling point and a high-boiling point should be in 10 - 90% of the weight of the range to the organic solvent whole quantity among organic solvents, respectively.

[0009] (b) The total content of an organic solvent should be in 1 - 50% of the weight of the range to the whole photosensitive glass paste.

[0010] (c) As an indispensable component of a photosensitive organic component, the oligomer or the polymer of the weight molecular weight 500-100,000 which has a partial saturation double bond should be contained in intramolecular ten to 90% of the weight to the amount of photosensitive organic substance at a carboxyl group or/and intramolecular.

[0011] (d) In the photosensitive organic component, organic dye should be contained 0.05 to 2% of the weight to the photosensitive organic substance.

[0012] Moreover, the manufacture approach of the plasma display of this invention The above-mentioned photosensitive glass paste is applied on a substrate using the above-mentioned photosensitive glass paste. It is the manufacture approach of a plasma display including the process which is pattern-exposed, develops negatives after desiccation, calcinates further, and forms a septum. It is characterized by setting 1 time of spreading thickness of

this photosensitive glass paste to 50 micrometers or more, and sets to this invention. It is desirable to apply this photosensitive glass paste that forms a septum by the doctor blade method or the slit-die coat method, and it can apply this photosensitive glass paste that forms a septum by 1 time or 2 times.

[0013]

[Embodiment of the Invention] In the photosensitive glass paste which consists of glass powder, a photosensitive organic component, and an organic solvent, this invention becomes able [climax of the edge section of a paste coating layer] to form few paste coating layers of uniform thickness after paste coating and desiccation, when these two or more kinds of organic solvents are contained and the boiling point difference of at least two kinds of organic solvents considers as 20 degrees C or more among these organic solvents. In this invention, glass powder, a photosensitive monomer, oligomer, a polymer, an organic solvent, the photopolymerization initiator, the sensitizer, the sensitization assistant, the plasticizer, etc. are usually contained in the photosensitive glass paste.

[0014] As an organic solvent used in this invention, the liquid whose boiling point in ordinary pressure is 20-250 degrees C is used suitably. It is the boiling point, volatility, solubility over the photosensitive organic substance, a miscibility, a distributed property, a rheology property, etc. in ordinary pressure that it must take into consideration in case the organic solvent to add is selected. For example, when the boiling point in ordinary pressure is not much high, it is necessary to make drying temperature high, and a photosensitive organic component pyrolyzes by it, or we are anxious about causing thermal polymerization. Moreover, when a solvent excellent in volatility is used, while there is an advantage that desiccation is easy, the leveling nature of a paste worsens, surface surface smoothness will be inferior and it becomes difficult further for solvent evaporation to stabilize a paste presentation. Moreover, when the solubility and dispersibility over the photosensitive organic substance are bad, the inconvenience of being unable to form the paste coating film of uniform thickness arises. The organic solvent to add is selected fully in consideration of these points.

[0015] As an organic solvent preferably used by this invention Gamma-butyrolactone (it omits gamma-BL below), and methyl cellosolve, ethylcellosolve, Cellosolve, such as butyl cellosolve, isopropyl alcohol, methyl alcohol, Alcohols, such as ethyl alcohol, butyl alcohol, and normal propyl alcohol, A methyl ethyl ketone, dioxane, an acetone, cyclopentanone, a diethyl ketone, Ketones, such as methyl isobutyl ketone and diisobutyl ketone, ethyl lactate and methyl acetate, Ethyl acetate, isopropyl acetate, normal propyl acetate, isobutyl acetate, normal pentyl acetate, and lo — pentyl acetate — 3-methoxy-3-methyl-1-butanol, 3-methoxy-3-methyl-butyl acetate, Ester, such as propylene glycol 1-monomethyl ether-2-acetate Chlorinated hydrocarbon, such as hydrocarbons, such as a hexane, a cyclohexane, benzene, toluene, and a xylene, a methylene chloride, chloroform, and dichloromethane, is mentioned.

[0016] As shown in the below-mentioned example 1 of a comparison among these organic solvents, the photosensitive glass paste which added only the gamma-butyrolactone (gamma-BL) whose boiling point in ordinary pressure is 200-208 degrees C is applied once with a doctor blade method, and if it is made to dry, the edge section of a paste coating layer (170 micrometers of thickness after desiccation) will cover 2cm of ****, and will rise about 30 micrometers. If it is going to expose using a mask in this condition and is going to flush the unexposed section of a climax part with a developer, an exposure part, i.e., a septum part, will corrode too much with a developer, and the serious problem of moving in a zigzag direction or separating will arise. That is, since developing time becomes various when paste thickness is uneven, a beautiful septum without a residual membrane, meandering, and peeling cannot be formed. Therefore, it is important for paste coating thickness to make it homogeneity as much as possible.

[0017] In operation of this invention, the need has making two or more kinds of organic solvents which have a boiling point difference in ordinary pressure as an organic solvent which is an indispensable component contain, and as for the boiling point difference in the ordinary pressure of the organic solvent by the side of those low-boiling points and a high-boiling point, it is desirable that it is the range of 20-100 degrees C, and it is the range of 30-60 degrees C more preferably. When there are few boiling point differences, about 30 micrometers of the amounts of climaxes of the spreading side edge section after desiccation cannot rise like the case where one kind of organic solvent is used, and they cannot reduce the amount of climaxes. Moreover, when there is a boiling point difference 100 degrees C or more, setting out of drying temperature becomes difficult. When drying temperature is set as the middle temperature of a high-boiling point solvent and a low-boiling point solvent, it is because a high-boiling point solvent will remain considerably. Moreover, when drying temperature is set as the temperature beyond a high-boiling point, a possibility that the photosensitive organic substance may cause a pyrolysis and thermal polymerization is large. Therefore, as for the boiling point difference in the ordinary pressure of an organic solvent, it is desirable that it will be the range of 20-100 degrees C if it can do, and it is 30-60 degrees C more preferably.

[0018] Moreover, the content to the whole photosensitive glass paste of an organic solvent is 10 - 40 % of the weight more preferably one to 60% of the weight. If the amount of an organic solvent increases too much, the drying time will become long and productivity will worsen.

[0019] Furthermore, the content of each organic solvent by the side of the low-boiling point whose boiling point difference is 20 degrees C or more, and a high-boiling point has 10 - 90% of the weight of the desirable range respectively to the organic solvent whole quantity, and is 30 - 70 % of the weight more preferably. When the number of organic solvents is two, and there are extremely few contents of any one kind of organic solvent, as a result, the effectiveness which added the solvent is not acquired and the amount of climaxes of the spreading layer edge section is not reduced. moreover, the thing which two kinds of organic solvents, a low-boiling point side and a high-boiling point side, are in the above-mentioned range in this invention, respectively when using three or more kinds of

organic solvents — desirable — other organic solvents — the above — it may be out of range. Or it is desirable that it classifies into a low-boiling point and high-boiling point side, and each organic solvent group is in the above-mentioned range.

[0020] The photosensitive organic substance in this invention means a photosensitive monomer, photosensitive oligomer, and photosensitive polymer. A photosensitive glass paste can be made to contain a photopolymerization initiator, a sensitizer, a sensitization assistant, polymerization inhibitor, a binder, a plasticizer, a RE ** ring agent, an ultraviolet ray absorbent, etc. if needed in addition to these photosensitivity organic substance.

[0021] As a photosensitive monomer, it is the compound which has a carbon-carbon unsaturated bond. Specifically Methyl acrylate, ethyl acrylate, n-propylacrylate, Isopropyl acrylate, n-butyl acrylate, sec-butyl acrylate, Isobutyl acrylate, t-butyl acrylate, n-pentyl acrylate, Allyl compound acrylate, benzyl acrylate, butoxy ethyl acrylate, Butoxy triethylene glycol acrylate, cyclohexyl acrylate, Cyclo cutting-pliers nil acrylate, cyclo pentenyl acrylate, 2-ethylhexyl acrylate, Glycerol acrylate, glycidyl acrylate, heptadeca FUROROJI sill acrylate, 2-hydroxyethyl acrylate, ISOBO nil acrylate, 2-hydroxypropyl acrylate, ISODEKI sill acrylate, iso octyl acrylate, laurylacrylate, 2-methoxy ethyl acrylate, methoxy ethylene glycol acrylate, Methoxy JIECHIRREN glycol acrylate, octaphloropentyl acrylate, FENIKISHI ethyl acrylate, stearylacrylate, triphloroethyl acrylate, Arylation HEKISHI diacrylate, 1,4-butanediol diacrylate, 1,3-butylene-glycol diacrylate, ethylene glycol diacrylate, Diethylene glycol diacrylate, triethylene glycol diacrylate, Polyethylene-glycol diacrylate, dipentaerythritol hexaacrylate, Dipentaerythritolmonohydroxypentaacrylate, ditrimethylolpropanetetraacrylate, Glycerol diacrylate, methoxy-ized cyclohexyl diacrylate, Neopentyl glycol diacrylate, propylene glycol diacrylate, Polypropylene-glycol diacrylate, triglycerol diacrylate, Trimethylolpropane triacrylate, acrylamide, aminoethyl acrylate, Phenyl acrylate, phenoxy ethyl acrylate, benzyl acrylate, 1-naphthyl acrylate, 2-naphthyl acrylate, bisphenol A diacrylate, Acrylate, such as diacrylate of the bisphenol A-ethyleneoxide addition product, thiophenol acrylate, and benzyl mercaptan acrylate, etc. can be mentioned.

[0022] The development nature after sensitization can be improved by adding partial saturation acids, such as unsaturated carboxylic acid, in addition to these. As an example of unsaturated carboxylic acid, an acrylic acid, methacrylic acid, an itaconic acid, a crotonic acid, a maleic acid, a fumaric acid, vinyl acetic acids, or these acid anhydrides are mentioned.

[0023] In this invention, the content of the monomer as these photosensitivity organic substance has 5 – 30 desirable % of the weight to the sum of glass powder and the photosensitive organic substance. In any range other than these, since there is an inclination which the lack of hardening after aggravation of pattern formation nature and hardening generates, it is not desirable.

[0024] Moreover, what compounded one or more kinds of the above-mentioned photosensitive monomer, and was obtained as the photosensitive oligomer which is the photosensitive organic substance, and photosensitive polymer can be used. The content of these photosensitivity oligomer and photosensitive polymer has 5 – 30 desirable % of the weight to the sum of glass powder and the photosensitive organic substance. In any range other than these, since a pattern is impossible, **** of a pattern arises or there is an inclination for meandering to become large, it is not desirable.

[0025] In this invention, it is desirable that the oligomer or the polymer of the weight molecular weight 500–100,000 which has a partial saturation double bond in a carboxyl group or/and intramolecular is contained in intramolecular ten to 90% of the weight to the amount of photosensitive organic substance as an indispensable component of a photosensitive organic component. The operating rate of a photosensitive organic component has 5 – 35 desirable % of the weight from the point of contraction after pattern formation nature and baking during the photosensitive glass paste of this invention. If out of range, since [this] pattern formation nature's being impossible or **** of a pattern arises, it is not desirable.

[0026] Moreover, as for the glass powder used by this invention, it is desirable that the coefficient of thermal expansion (α_{50-400}) of 50–400 degrees C is 50 to 90×10^{-7} . Moreover, when oxidation silicon blends and boron oxide blends in 5 – 50% of the weight of the range three to 60% of the weight into glass, the electrical and electric equipment, a machine, and thermal properties, such as the compactness of electric insulation, reinforcement, a coefficient of thermal expansion, and an insulating layer, can be improved. It is desirable that glass transition temperature is 430–500 degrees C, and softening temperature is 470–580 degrees C. It is because it must calcinate at an elevated temperature and distortion will arise in a substrate in the case of baking, if 500 degrees C and softening temperature have a glass transition temperature higher than 580 degrees C. Moreover, in the case of glass with a lower softening temperature [430 degrees C and softening temperature] than 470 degrees C glass transition temperature, a precise septum layer is not obtained but it becomes the cause of peeling of a septum, an open circuit, and meandering. Although glass powder particle diameter is chosen in consideration of the line breadth and the height of a septum which it is going to produce, it is desirable that 50 volume % particle diameter (mean particle diameter D50) is 1–6 micrometers, and grain-of-maximum-size sizes are 30 micrometers or less, and specific surface area of $1.5-4 \text{ cm}^2 / \text{g}$. As a photopolymerization initiator, a benzophenone, methyl o-benzoylbenzoate, 4 and 4-screw (dimethylamine) benzophenone, 4, and 4-dichloro benzophenone, 4-benzoyl-4-methyl diphenyl ketone, dibenzyl ketone, full — me — non, 2, and 2-dimethoxy aceti phenon, 2, and 2-dimethoxy-2-methyl-2-phenyl-2-phenyl aceti phenon — 2-hydroxy-2-methylpropiohenone, a p-t-butyl dichloro acetophenone, A thioxan ton, 2-methyl CHOO xanthone, 2-chloro thioxan ton, 2-isopropyl thioxan ton, a diethyl thioxan ton, benzyl, Benzyl dimethyl methyl KETANORU, a benzyl methoxy ethyl acetal, A benzoin, benzoin methyl ether, benzoin butyl ether, Anthraquinone, 2-t-butyl anthraquinone, 2-aminoanthraquinone, beta-KURORU anthraquinone, an anthrone,

benzanthrone, dibenzosulfone, A methylene anthrone, 4-azide benzal acetophenone, 2, 6-screw (P-azide benzylidene) cyclohexanone, A 2-phenyl -1, 2-butanedione 2-(o-methoxycarbonyl) oxime and 1, and 3-diphenylpropane trione-2-(o-ethoxycarbonyl) oxime etc. is mentioned. In this invention, one or more kinds of these can be used. This photopolymerization initiator is 0.1 - 15% of the weight of the range preferably [adding in 0.05 - 20% of the weight of the range to a photosensitive component], and more preferably. If there are too few amounts of an initiator, photosensitivity will fall and it will become the lack of hardening. On the other hand, a septum grows fat when many [too].

[0027] As an example of a sensitizer, 2, 4-diethyl thioxan ton, an isopropyl thioxan ton, 2, 3-screw (4-dimethylamino benzal) cyclohexanone, 2, 6-screw (4-dimethylamino benzal)-4-methylcyclohexanone, A Michler's-ketone, 4, and 4-screw (diethylamino)-benzophenone, 4 and 4-screw (dimethylamino) chalcone, 4, and 4-screw (diethylamino) chalcone, p-dimethylamino thinner millimeter DENIMIDANON, p-dimethylamino benzyl DIN INDANONNA, etc. are mentioned. These one or more kinds can be used in this invention. An addition is 0.1 - 10 % of the weight more preferably 0.05 to 10% of the weight to a photosensitive component. Photosensitivity cannot be raised when there are few amounts of a sensitizer. On the other hand, when many [too], photosensitivity becomes sensitive too much and a septum grows fat.

[0028] Polymerization inhibitor is added in order to raise preservation stability. As an example, the monoester compound of a hydroquinone and a hydroquinone, an N-nitroso diphenylamine, phenothiazin, a p-t-butyl catechol and N-N phenyl-naphthylamine, clo RANIRU, etc. are mentioned. The addition is usually 0.001 - 1 % of the weight.

[0029] As a binder, polyvinyl alcohol, a polyvinyl butyral, a methacrylic ester polymer, an acrylic ester polymer, an acrylic-acid ester methacrylic ester polymer, an alpha-methyl-styrene polymer, butyl methacrylate resin, etc. are mentioned. Dibutyl phthalate, dioctyl phthalate, a glycerol, etc. are mentioned as an example of a plasticizer.

[0030] Moreover, in this invention, it is desirable to add an ultraviolet ray absorbent and a high definition and high resolution are obtained. As an ultraviolet ray absorbent, the organic system color is used well. Specifically, an azo system color, an amino ketone system color, a xanthene system color, a quinoline system color, an amino ketone system color, an anthraquinone system color, a BENO phenon system color, etc. can be used. The addition has 0.05 - 5 desirable % of the weight to glass powder. If many [if there are few additions, the addition effectiveness of an ultraviolet ray absorbent will decrease, and / too], the insulator layer property after baking falls and is not desirable. It is 0.05 - 0.18 % of the weight more preferably.

[0031] An example of the addition approach of an optical absorption agent which consists of organic dye in this invention is given. Organic dye is dissolved in organic solvents, such as an acetone, and it mixes with inorganic powder, and fully agitates. Next, an organic solvent is evaporated using a rotor RIEBA porter. The so-called powder of the shape of a capsule which carried out the coat of the film of organic dye to each powder front face of a non-subtlety particle by this approach is producible.

[0032] An antioxidant is added in order to prevent oxidation of the acrylic copolymer at the time of preservation. 2, 6-G t-p-cresol, BUCHIRUKA hydroxylanisole, 2, 6-G t-4-ethylphenol, and 2,2-methylene bis - (4-methyl-6-t-butylphenol) etc. is mentioned as an example. An addition is usually 0.001 - 1 % of the weight to a paste.

[0033] In the activity of the photosensitive glass paste of this invention, although, as for after paste coating, desiccation is naturally needed, the notes at the time of this desiccation are keeping a *****-strike level. It is because a *-strike flows and thickness unevenness occurs, when leaning. Although approaches generally used well, such as hot blast oven, a hot plate, far infrared rays, air drying, and reduced pressure drying, can be used as the desiccation approach, an important point is a point of making homogeneity dry a *****-strike layer to the depth direction. For example, since heat transmits to the upper part from a paste pars basilaris ossis occipitalis and a paste pars basilaris ossis occipitalis dries ahead of a front face when it dries using a hot plate, there is no possibility of blowing air bubbles. An air drying requires time amount and its productivity is bad. Moreover, a decompression device is needed, reduced pressure drying requires an installation cost, and its productivity is also bad. As for desiccation of such a point to a photosensitive glass paste, it is desirable to use a hot plate.

[0034] Although what times are sufficient as drying temperature as long as the photosensitive organic substance is temperature lower than the temperature which does not cause thermal polymerization, generally its range of 40-150 degrees C is desirable. In the desiccation using a hot plate, in the case of hot blast oven, drying temperature means the thing of the temperature of hot blast for the thing of the temperature on the front face of a hot plate here.

[0035] Next, the preparation approach of the photosensitive glass paste of this invention is explained. After preparing glass powder, a photosensitive monomer, oligomer and a polymer, a photopolymerization initiator, a sensitizer, and the various components of an organic solvent so that it may become a predetermined presentation, with 3 rollers or a kneading machine, to homogeneity, a photosensitive glass paste is mixed and distributed and usually produces them. Although the viscosity of this paste is decided by addition rates, such as glass powder, a photosensitive monomer, oligomer, a polymer, an organic solvent, and a plasticizer, that range is 0.5-200Paands preferably. For example, spreading to a glass substrate has 0.5-2Pa and s desirable at 2 - 50 Pa-s and a spin coat method in screen printing by 30-200Paands, the doctor blade method, or the slit-die coat method.

[0036] Next, an example which performs pattern processing using a photosensitive glass paste is explained. A photosensitive glass paste is applied to complete spreading or a partial target on a glass substrate, a ceramic substrate, or the film made from a polymer. As the method of application, general approaches, such as screen-stencil, a bar coat, a roll coat, a slit-die coat, a doctor blade, and a spin coat, can be used.

[0037] Here, when applying a photosensitive glass paste on a substrate, in order to raise the adhesion of a substrate and the spreading film, a substrate can be beforehand processed with surface treatment liquid. As surface-

preparation liquid, silane coupling, vinyl trichlorosilane, vinyltrimethoxysilane, vinyltriethoxysilane, tris-(2-methoxyethoxy) vinylsilane, gamma-glycidoxy propane trimethoxysilane, gamma-(methacryloxypropyl) trimethoxysilane, gamma-(2-aminoethyl) aminopropyl trimethoxysilane, gamma-chloropropyltrimethoxysilane, gamma-mercaptopropyltrimethoxysilane, etc. are an organic metal, for example, organic titanium, or organic aluminum, an organic zirconium, etc. What diluted the silane coupling agent or the organic metal with an organic solvent, for example, ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, methyl alcohol, ethyl alcohol, propyl alcohol, etc. to 0.5 - 5% of concentration is used. Next, after applying this surface treatment liquid to homogeneity on a substrate with a spinner etc., surface treatment can be carried out by drying at 80-140 degrees C for 10 to 60 minutes.

[0038] It exposes after spreading of a photosensitive glass paste using an aligner. The approach of carrying out mask exposure using a photo mask is common so that exposure may be performed by the usual photolithography. The mask used selects either a negative mold or a positive type according to the class of photosensitive organic component. moreover, a photo mask — using — ruble — there are nothings and the approach of carrying out direct writing by red, a blue laser beam, etc. may be used. As an aligner, a stepper exposure machine, a pro squeak tee exposure machine, etc. can be used. Moreover, when exposing a large area, a large area can be exposed in a small exposure area by exposing conveying, after applying a photosensitive paste on substrates, such as a glass substrate. In this case, as the activity light source used, although a visible ray, a near ultraviolet ray, ultraviolet rays, an electron ray, an X-ray, a laser beam, etc. are raised, also in these, ultraviolet rays are desirable and a low pressure mercury lamp, a high-pressure mercury-vapor lamp, an ultrahigh pressure mercury lamp, a halogen lamp, germicidal lamp glass, etc. can be used as that light source, for example. An ultrahigh pressure mercury lamp is suitable also in these. Although exposure conditions change with spreading thickness, it is desirable to expose for 0.2 - 30 minutes using the ultrahigh pressure mercury lamp of the output of 1 - 100 mW/cm².

[0039] In the applied photosensitive glass paste front face, a pattern configuration can be raised by preparing the oxygen cutoff film. As an example of the oxygen cutoff film, films, such as film, such as PVA and a cellulose, or polyester, are raised. After concentration applies to homogeneity on a substrate the water solution which is 0.5 - 5 % of the weight by the approach of a spinner etc., by drying for 10 - 60 minutes at 70-90 degrees C, the formation approach of the PVA film evaporates moisture and is performed. Moreover, since it will be smeared as an insulator layer, a sex will become good and evaporation will become easy if little addition of the alcohol is carried out into a water solution, it is desirable. Still more desirable PVA solution concentration is 1 - 3 % of the weight, and if it is in this range, it can raise sensibility further. The reason whose sensibility improves by PVA spreading is presumed as follows. That is, if there is oxygen in air in case a photosensitive component carries out the photoreaction, it will be thought that the sensibility of photo-curing is checked, but since excessive oxygen can be intercepted if there is film of PVA, it is thought that sensibility improves at the time of exposure. When using transparent films, such as polyester, and polyprene, polyethylene, there is also a method of sticking and using these films after the photosensitive glass paste after spreading.

[0040] Although negatives are developed after exposure using the difference of the solubility to the development of a sensitization part and a non-exposing part, development is performed by dip coating, the spray method, and the brush method. The organic solvent which can dissolve the organic component under photosensitive glass paste can be used for the developer used. Moreover, water may be added in the range in which the solvent power is not lost in the organic solvent. When the compound which has acidic groups, such as a carboxyl group, during a photosensitive glass paste exists, negatives can be developed in an alkali water solution. Although metal alkali water solutions, such as a sodium hydroxide, a sodium carbonate, and a calcium-hydroxide water solution, are used as an alkali water solution, since having used the organic alkali water solution tends to remove an alkali component at the time of baking, it is desirable. An amine compound can be used as organic alkali. Specifically, tetramethylammonium hydroxide, trimethyl benzyl ammonium hydroxide, monoethanolamine, diethanolamine, etc. are mentioned. The concentration of an alkali water solution is usually 0.1 - 5 % of the weight more preferably 0.01 to 10% of the weight. A fusible part is not removed when alkali concentration is too low. Moreover, it is [a possibility of making the pattern section exfoliating and making a non-fusible part corroding] and is not desirable when alkali concentration is too high. Moreover, as for the development temperature at the time of development, it is desirable on production control to carry out at 20-50 degrees C.

[0041] Next, it calcinates with a firing furnace. Although a firing environments and temperature change with classes of substrate of a paste, they are calcinated in ambient atmospheres, such as nitrogen and hydrogen, among air. As a firing furnace, the continuation mold firing furnace of belt types, such as a firing furnace of a batch type and a roller HASU mold, can be used. Burning temperature is 500-610 degrees C in temperature, and calcinates by holding for 5 - 60 minutes. Especially desirable temperature is 530-580 degrees C.

[0042] Moreover, 50-200-degree-C heating process may be introduced for the object of desiccation and a preliminary reaction into each process of above-mentioned spreading and exposure, development, and baking. When manufacturing a plasma display, on the glass substrate in which the electrode layer was formed, the photosensitive glass paste of this invention is used, a septum is formed according to the above-mentioned process, a fluorescent substance can be further formed with screen printing or photosensitive glass mull technique, and a tooth-back substrate can be obtained. The tooth-back substrate and front substrate which were obtained are set, and closure and after carrying out rare-gas installation, a plasma display can be manufactured by connecting an actuation circuit.

[0043]

[Example] Hereafter, this invention is concretely explained using an example. However, this invention is not limited to this. In addition, especially the concentration in an example and the example of a comparison is weight % unless it refuses.

[0044] (Example 1) Following glass particles and the following following organic component were added at a following paste presentation rate, and the photosensitive paste was produced. It dissolved first, heating each component of an organic component at 80 degrees C, and the production procedure created the paste by adding glass particles and kneading with a kneading machine after that.

[0045] Climax of the edge section measured by scan-speed 0.6 mm/s using the surface roughness meter (surfboard COM 1500A Tokyo Seimitsu Science). Moreover, electron microscope observation performed assessment of a septum pattern. The valuation basis made the case where a good configuration (the way of the lower part is trapezoidal shape with wide width of face for a while) was acquired as O, and moved in a zigzag direction, it separated, and it made x the case where the residual membrane remained.

[0046] Glass powder: Li₂O:8.6% and SiO₂:20.1%, B₂O₃:31%, BaO:3.8%, and aluminum₂O₃:20.6%, ZnO:2.1%, MgO:5.9%, and CaO:4.2% presentation (analysis value). The average refractive index was 1.586. Moreover, this glass powder carried out coating processing, and used the ultraviolet-rays extinction agent.

[0047]

A photosensitive monomer (MGP-400) : X₂-N-CH(CH₃)-CH₂-(O-CH₂-CH (CH₃))_n-N-X₂ X: -CH₂-CH(OH)-CH₂-O-CO-C=CH₂ n:2 - 10 photosensitive polymer (X-4007): 40% chestnut [meta-] acid, 30% methyl meta-KURIRE - TO, mosquito of the copolymer which consists of styrene 30% As opposed to a RUBOKISHIRU radical 0.4Eq GURISHIJI Weighted mean to which the addition polymerization of the RUMETA chestnut rate was carried out Photosensitive Pori of molecular weight 43,000 and the acid number 95 Ma.

[0048]

Photopolymerization initiator (IC-369): Irgacure-369 (Ciba-Geigy) shrine 2-benzyl-2-dimethylamino -1 - (4-morpholino phenyl) butanone - one sensitizer (DETX-S): — 2 and 4-diethyl thioxan ton ultraviolet-rays extinction agent (Sudan): — azo system color C24H20N4O organic solvent: — gamma-butyrolactone (200–208 degrees C of boiling points)

: Ethylcellosolve (135.6 degrees C of boiling points) The calculated value of the average refractive index of these organic components was about 1.59, and this was very close to the refractive index 1.586 of glass. A paste presentation Glass powder : 51.90% Photosensitive monomer : 11.64% Photosensitive polymer : 9.46% photopolymerization initiator : 2.60% A sensitizer : 2.60% Ultraviolet-rays extinction agent : 0.10% Organic solvent : Gamma-butyrolactone 10.85% Ethylcellosolve It is a doctor blade method on 10.85%, next 100mm angle glass substrate. After applying the above-mentioned photosensitive glass paste and making it dry for 120 minutes at 80 degrees C in path clearance 420micrometer using a hot plate, they were **, alias *****, and 2 micrometers, about climax of the edge section of the paste coating film.

[0049] Next, mask exposure was performed. The chromium mask designed so that the septum pattern formation of the shape of a stripe in pitch 220micrometer, the line breadth of 60 micrometers, and a plasma display might become possible as a mask was used. Exposure performed 0.8 J/cm² ultraviolet-rays exposure with the ultrahigh pressure mercury lamp with the output of 30 mW/cm². Then, shower development was performed at 35 degrees C using 0.3% water solution of monoethanolamine, and it dried for 10 minutes at 80 degrees C. A result is shown below.

[0050] Climax of the edge section: Height of 2 micrometer septum: 179-micrometer half-value width: 63-micrometer configuration: Gamma-butyrolactone (200–208 degrees C of boiling points) and ethyl lactate (154 degrees C of boiling points) were used as an organic solvent as the presentation of 0 (example 2) degree, and also it carried out like the example 1.

[0051]

A paste presentation Glass powder : 51.90% Photosensitive monomer : 11.64% Photosensitive polymer : 9.46% photopolymerization initiator : 2.60% sensitizer : 2.60% Ultraviolet-rays extinction agent : 0.10% Organic solvent : Gamma-butyrolactone 15.85% Ethyl lactate 5.85% result It is shown below.

[0052] climax of the edge section: height [of 3 micrometer septum]: — 181-micrometer half-value width: 60-micrometer configuration: O (example 3) — gamma-butyrolactone (200–208 degrees C of boiling points) and 3-methoxy-3-methyl-1-butanol (174 degrees C of boiling points) were used as an organic solvent as the following presentation, and also it carried out like the example 1.

[0053]

A paste presentation Glass powder : 51.90% Photosensitive monomer : 11.64% Photosensitive polymer : 9.46% photopolymerization initiator : 2.60% A sensitizer : 2.60% Ultraviolet-rays extinction agent : 0.10% Organic solvent : Gamma-butyrolactone 16.00% 3-methoxy-3-methyl - 1-butanol 5.70% result It is shown below.

[0054] Climax of the edge section: Height of 3 micrometer septum: 180-micrometer half-value width: 62-micrometer configuration: The paste of a presentation of the following which made the photosensitive glass paste which transposed all the ethylcellosolve of the O (example 1 of comparison) example 1 to gamma-butyrolactone, i.e., an organic solvent, one kind of gamma-butyrolactone was created, and this **** for a photosensitive glass paste etc. was carried out like the example 1.

[0055]

A paste presentation Glass powder : 51.90% Photosensitive monomer : 11.64% Photosensitive polymer : 9.46% photopolymerization initiator : 2.60% A sensitizer : 2.60% Ultraviolet-rays extinction agent: 0.10% Organic solvent :

Gamma-butyrolactone Climax of the edge section of after 21.70% desiccation and the paste coating film They were **, alias *****, and 30 micrometers. Next, exposure and development were performed. When it was going to flush the paste of the unexposed section of the edge section with the developer, the septum part moved in a zigzag direction for too much corrosion to the septum part of a developer.

[0056] Climax of the edge section: Height of 30 micrometer septum: 172-micrometer half-value width: 65-micrometer configuration: x (meandering)

(Example 2 of a comparison) Created the photosensitive glass paste of the following presentation which changed the ethylcellosolve of an example 1, and the rate of gamma-butyrolactone as follows, namely, lessened the amount of ethylcellosolve, and this was used, and also it carried out like the example 1.

[0057]

A paste presentation Glass powder : 51.90% Photosensitive monomer : 11.64% Photosensitive polymer : 9.46% photopolymerization initiator : 2.60% A sensitizer : 2.60% Ultraviolet-rays extinction agent: 0.10% Organic solvent : Gamma-butyrolactone 20.00% Ethylcellosolve Climax of the edge section of after 1.70% desiccation and the paste coating film They were **, alias *****, and 28 micrometers. Next, when negatives tended to be developed by having exposed and it was going to flush the paste of the unexposed section of the edge section with the developer, the septum part moved in a zigzag direction.

[0058] Climax of the edge section: Height of 28 micrometer septum: 174-micrometer half-value width: 63-micrometer configuration: x (meandering)

[0059]

[Effect of the Invention] According to this invention, in the photosensitive glass paste which consists of glass powder, a photosensitive organic component, and two or more kinds of organic solvents, when these two or more kinds of organic solvents are contained and the boiling point difference of at least two kinds of organic solvents considers as 20 degrees C or more among these organic solvents, the photosensitive glass paste which enables climax of the spreading side edge section to form the paste coating film of little uniform thickness is obtained.

[0060] If it is in the condition in which the edge section of the paste coating film rose, it is specifically going to expose using a mask and it is going to flush the unexposed section of a climax part with a developer, by an exposure part's, i.e., a septum part's, corroding too much with a developer, and using the photosensitive glass paste of this invention, although the serious problem of moving in a zigzag direction or separating may arise, such a problem can be avoid and the quality substrate for plasma displays and a quality plasma display can be manufacture.

[Translation done.]